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SOLUBILITIES OF MOLYBDENUM AND TUNGSTEN IN LIQUID POTASSIUM

by Stephan Stecura

Lewis Research Center

Cleveland, Ohio



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16. Abstract A study was made of the solubilities of molybdenum and tungsten in liquid potassium containing less than 15 ppm by weight of oxygen. The solubility of molybdenum at 1040 and 1316 K is about 6 and 13 ppm by weight and that of tungsten at 1055 and 1328 K is about 20 and 130 ppm by weight, respectively. The apparent heats of solutions for molybdenum and tungsten solubilities in liquid potassium are 6.7 ± 0.8 and 17.6 ± 1.2 kcal/mole (28.0 and 73.7 kJ/mole), respectively. The solubility data for molybdenum are either the true solubility data, or relatively close to the true solubility data, for molybdenum in liquid potassium between 1040 and 1316 K. The solubility data for tungsten in liquid potassium between 1055 and 1328 K suggest that the solubility values might have been influenced by interstitial impurities, particularly by oxygen.			
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SOLUBILITIES OF MOLYBDENUM AND TUNGSTEN IN LIQUID POTASSIUM

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SUMMARY

The solubilities of molybdenum and tungsten in high-purity liquid potassium between 1040 and 1328 K were studied. Potassium used in this study contained less than 15 ppm by weight of oxygen. Molybdenum and tungsten solubilities in liquid potassium are described by the relations

$$\log \text{ wt ppm Mo} = (2.21 \pm 0.13) - \frac{(1472 \pm 156)}{T}$$

$$\log \text{ wt ppm W} = (5.02 \pm 0.21) - \frac{(3851 \pm 243)}{T}$$

where T is the absolute temperature. The heats of solutions for molybdenum and tungsten are 6.7 ± 0.8 and 17.6 ± 1.2 kilocalories per mole (28.0 and 73.7 kJ/mole), respectively.

The solubility data for molybdenum are either the true solubility data, or relatively close to the true solubility data, of molybdenum in liquid potassium between 1040 and 1316 K. The solubility data for tungsten in liquid potassium between 1055 and 1328 K suggest that the solubility values might have been influenced by the interstitial impurities, particularly by the oxygen.

INTRODUCTION

Alkali metals such as sodium, potassium, and lithium are of great interest as heat-transfer and thermodynamic working fluids. Potassium has been considered for use in advanced Rankine-type space-power systems up to 1473 K (refs. 1 and 2). At such high temperatures, alloys of refractory metals (tantalum, niobium, molybdenum, and tung-

sten) are needed for the containment of potassium or other alkali metals.

In studies of the compatibility of liquid alkali metals with potential containment materials, solubility data are needed to properly interpret the results of corrosion tests and also to assist in predicting corrosion behavior under test conditions that have not as yet been studied. Solubility data have been reported for the following refractory metals in liquid potassium: tantalum (refs. 3 and 4), niobium (refs. 3 and 5), molybdenum (refs. 3 and 5), tungsten (ref. 6), and vanadium (ref. 7).

There is a considerable disagreement and scatter in the reported solubility data, which may be attributed to the effects of interstitial impurities (O, N, and C). Recently published solubility and corrosion data (refs. 5, and 7 to 10) indicate that the solubilities of refractory metals are most significantly affected by the presence of oxygen. Nitrogen and carbon are believed to have lesser effects than oxygen.

The purpose of this investigation was to study the solubilities of molybdenum and tungsten in high-purity liquid potassium at high temperatures. The solubilities of these two refractory metals in liquid potassium between 1040 and 1328 K were determined by a rotatable-capsule method previously described in references 7 and 8.

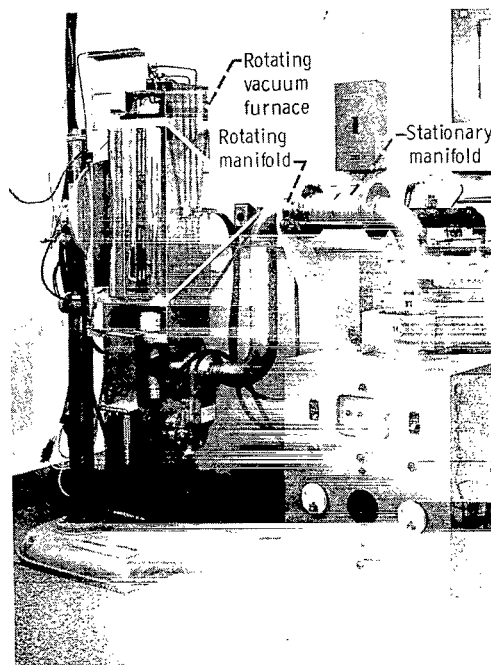
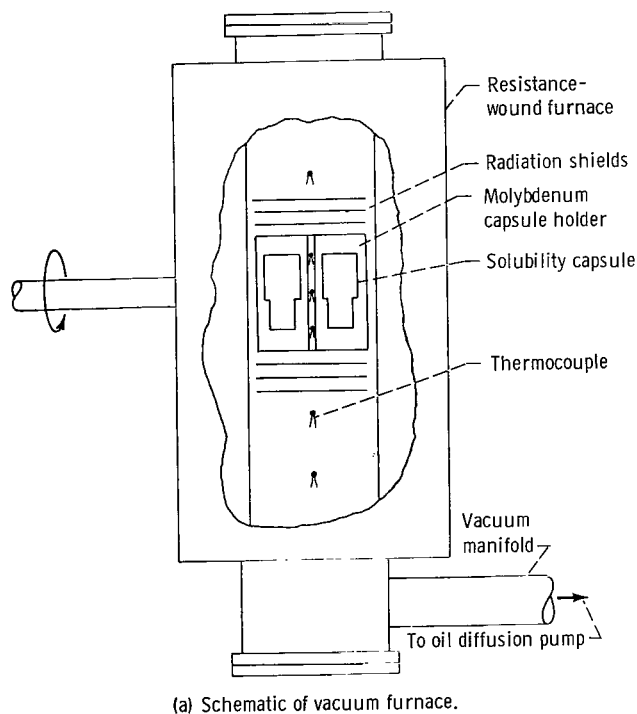


Figure 1. - Solubility apparatus.

MATERIALS, APPARATUS, AND PROCEDURE

The equipment used in this study is the same as that described in references 7 and 8 and shown in figure 1. The solubilities of molybdenum and tungsten in liquid potassium were determined by allowing the crucibles (source of solute) of these two metals to be in contact with liquid potassium for 24 hours at the desired temperature. The potassium and the crucibles were sealed in nickel capsules. After 24 hours at the desired temperature, the capsules were inverted and the potassium containing the solutes was allowed to flow from the crucibles into the empty parts of the capsules. The furnace was then cooled to room temperature, the capsules opened, and the potassium removed for analysis.

Materials

The potassium used in this study contained less than 15 ppm by weight of oxygen as determined by the vacuum amalgamation method.

The molybdenum and tungsten crucibles were machined from 1/2-inch (1.27-cm) diameter polycrystalline rods. Nickel capsules were machined from 5/8-inch (1.59-cm) diameter polycrystalline rod. The capsules were used as the containers for the crucibles and potassium, and as the collectors for the solute-containing potassium at the end of the test runs. The dimensions and the relative positions of the crucibles and capsules during a test run are given in figure 2.

The crucibles and capsules were cleaned and then analyzed for impurities. After they were ultrasonically cleaned in a fluorinated hydrocarbon, they were ultrasonically cleaned

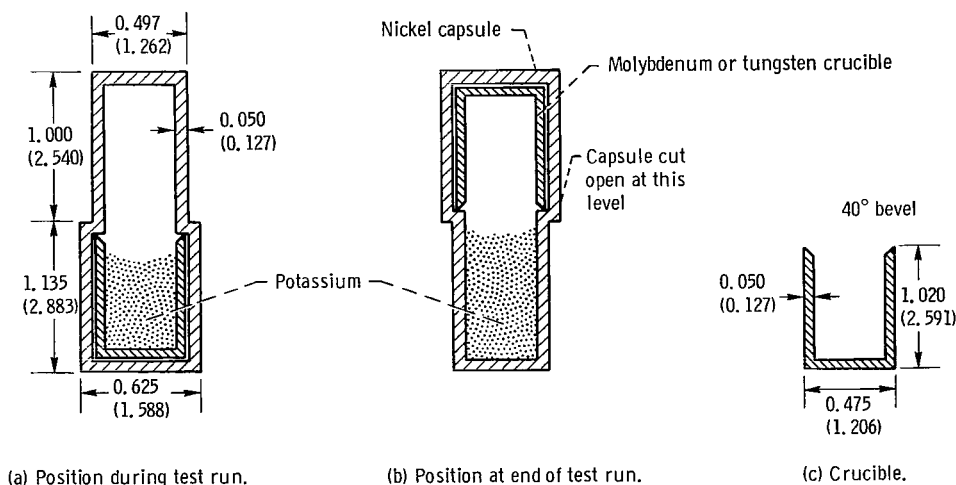


Figure 2. - Solubility capsule and crucible. Dimensions are in inches (cm).

TABLE I. - IMPURITY CONTENT OF CRUCIBLE AND
CAPSULE METALS

Impurity	Molybdenum	Tungsten	Nickel
Concentration of impurity, ppm by weight			
Carbon ^a	27	4	111
Oxygen ^a	10	5	13
Nitrogen ^a	7	3	18
Hydrogen ^a	1	1	4
Aluminum	1	20	5
Arsenic	(b)	<100	<1
Barium	<1	<1	<4
Beryllium	↓	↓	<1
Boron	↓	↓	<1
Cadmium	↓	↓	1
Calcium	↓	1	2
Cesium	(b)	1	<1
Chromium	<1	5	5
Cobalt	↓	<1	5
Copper	↓	<1	<1
Gallium	↓	(b)	<1
Germanium	↓	(b)	<2
Hafnium	(b)	(b)	<3
Iron	2	5	10
Lead	<1	<1	<1
Lithium	↓	↓	<1
Magnesium	↓	↓	<1
Manganese	↓	↓	2
Molybdenum	High	5	<1
Nickel	<1	5	High
Potassium	<1	1	<1
Rubidium	<1	1	<1
Silicon	5	1	<1
Silver	<1	<1	3
Sodium	<1	<1	<1
Strontium	<1	<1	<5
Tellurium	(b)	<100	<1
Tin	<1	<1	<2
Titanium	1	3	8
Tungsten ^a	(b)	High	<1
Vanadium	<1	<1	<1
Zinc	<10	<10	6
Zirconium	(b)	<1	<2

^aResults obtained by standard analytical measurements.

Molybdenum and tungsten data obtained by optical spectrographic method. Nickel data obtained by mass spectrographic method.

^bNot detected.

in a detergent. Then the crucibles and the capsules were etched. The etchant solutions and the procedures are given in the appendix.

Analyses of impurities were done on randomly selected samples of cleaned crucibles and capsules. Two crucibles of each of the two solute metals and two nickel capsules were selected. As indicated in table I, the total impurity in molybdenum did not exceed 0.01 weight percent and the total impurities in tungsten and nickel were not greater than 0.03 weight percent. The impurities in the crucibles and the capsules were determined by standard analytical methods.

Apparatus

The solubility furnace used in this study (fig. 1) was the same as that described in reference 7. The largest temperature variation along the capsule walls, inside the 4-inch (10.16-cm) long molybdenum holder over a period of 24 hours, was $\pm 4^{\circ}$ at 1328 K. Five platinum - platinum-13 percent rhodium thermocouples, spaced about $1\frac{1}{2}$ inches (3.81 cm) apart, were used to measure the temperature. The estimated accuracy of the temperature measurement is about $\pm 8^{\circ}$ at 1328 K.

At the end of each test run, the furnace is inverted. The rotation of the furnace into the inverted position is necessary because the potassium containing molybdenum or tungsten in solution must be collected in the nickel capsule. The solute that precipitates during cooling of the furnace to room temperature can then be recovered from the capsule for analysis.

Procedure

The experimental technique used in this study was the same as that described in reference 7. Nickel capsules were used in determining the solubilities of molybdenum and tungsten in liquid potassium because high-purity nickel metal has low concentrations of oxygen and nitrogen (table I) and it can easily be machined into capsules. Solute crucibles were made from molybdenum and tungsten metals.

The handling of potassium and the welding of the capsules were carried out in a vacuum chamber at about 5×10^{-6} torr pressure. Each capsule was filled with 1.2 ± 0.04 grams of potassium and the crucibles were then placed into the capsules. The capsules were capped, electron beam welded, and then leak checked.

The tests were run in the vacuum furnace (fig. 1) at about 2×10^{-6} torr pressure. At the end of each test run, the furnace was inverted. After cooling to room temperature,

the capsules were removed from the furnace and cut open near the middle of the capsule (fig. 2). The potassium containing the solutes was dissolved in butyl alcohol. The inside wall of the smaller-diameter capsule section was leached with hydrochloric acid to remove the solute that precipitated on cooling. Butyl alcohol and acid solutions were combined. Potassium in the combined solution was converted to potassium chloride by acidifying the combined solution with hydrochloric acid and evaporating the solvent. The weight of potassium chloride was determined gravimetrically after ignition at 723 K. The concentrations of molybdenum and tungsten in the potassium chloride samples were determined by spectrophotometric methods (refs. 11 and 12).

RESULTS

Highly reproducible solubility data were obtained for high-purity molybdenum and tungsten metals in liquid potassium. Each of these refractory metals had a total purity of about 99.97 percent (table I).

The solubilities of molybdenum and tungsten in high-purity liquid potassium between 1040 and 1328 K were studied. The results presented in table II and figures 3(a) and (b) were obtained by standard analytical methods (refs. 11 and 12). The accuracies of the analytical methods for the determinations of molybdenum and tungsten concentrations in the potassium chloride samples were determined by adding known amounts of the standard materials to the samples. The estimated accuracies of the analytical methods are ± 10 percent.

Molybdenum and tungsten solubility data reported in table II and figure 3 can be represented by the relations

$$\log \text{ wt ppm Mo} = (2.21 \pm 0.13) - \frac{(1472 \pm 156)}{T}$$

$$\log \text{ wt ppm W} = (5.02 \pm 0.21) - \frac{(3851 \pm 243)}{T}$$

where T is the absolute temperature. These two mathematical relations were derived by the least-squares method. In the derivation of the mathematical relation for the solubility of molybdenum in liquid potassium, one of the points at 1040 K (fig. 3(a)) was not used in the calculation. This point was considered a statistical "outlier" since it was outside the 3-sigma (standard deviation) limit.

The apparent heats of solutions for the molybdenum and tungsten solubilities in high-purity liquid potassium were calculated to be 6.7 ± 0.8 and 17.6 ± 1.2 kilocalories per mole (28.0 and 73.7 kJ/mole), respectively.

TABLE II. - SOLUBILITY DATA FOR MOLYBDENUM AND
TUNGSTEN IN HIGH-PURITY LIQUID POTASSIUM

Temperature, K	Molybdenum, ppm by weight	Temperature, K	Tungsten, ppm by weight
1316	14; 12	1328	125; 115
1269	12; 10	1287	135; 99
1207	10; 9	1207	80; 65
1176	9; 8	1182	60
1095	8; 7	1124	44; 34
1040	11; 6; 6	1069	30; 25
		1055	21

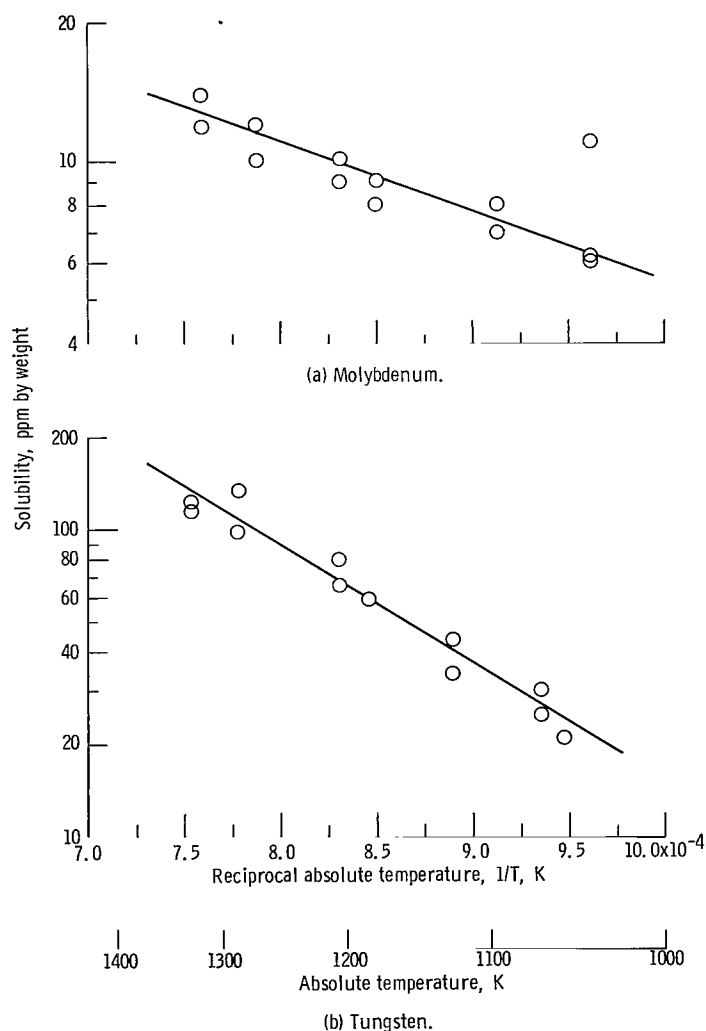


Figure 3. - Solubility of molybdenum and tungsten in high-purity potassium.

DISCUSSION

In evaluating the solubility data of refractory metals in liquid potassium, one must consider the magnitudes of the solubility and the heats of solution values in conjunction with the possible effect of oxygen on the solubilities. The low solubility values for molybdenum in liquid potassium (table II and fig. 3(a)) measured in this study are basically in agreement with the data reported by McKisson and others (ref. 3). However, the apparent heats of solutions calculated from these two sets of data are significantly different. The apparent heat of solution reported by McKisson and others (ref. 3) is about 25.6 kilocalories per mole (107 kJ/mole) as compared to 6.7 kilocalories per mole (28.0 kJ/mole) reported in this study. The author believes that this difference in the apparent heats of

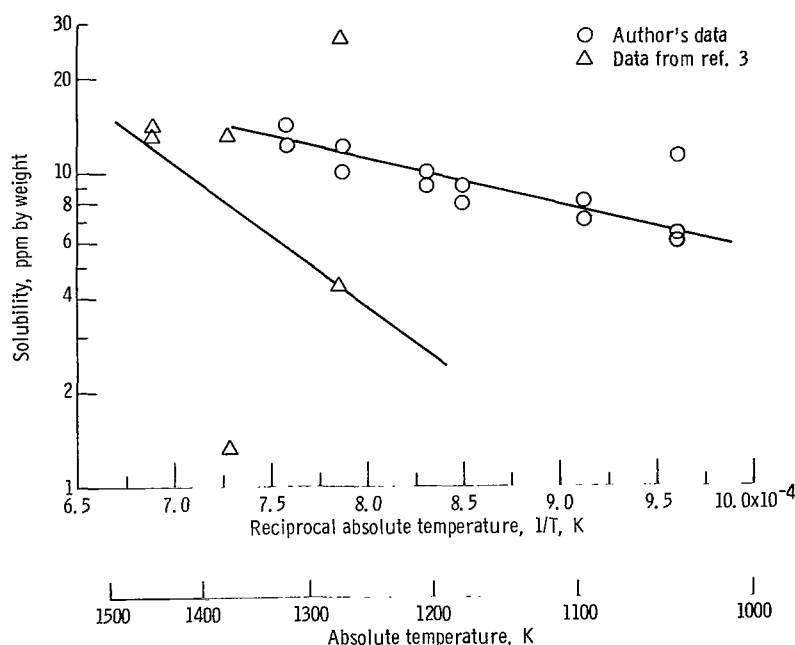


Figure 4. - Comparison of solubility data for molybdenum in liquid potassium.

solutions might be due to the poor reproducibility in the data (see fig. 4) reported by McKisson and others (ref. 3).

The effect of oxygen on the solubility of molybdenum was not investigated in this study. However, the effect of oxygen on the solubility of molybdenum in liquid potassium was studied by Cleary and others (ref. 5). The data reported by Cleary and others show that the solubility of molybdenum in liquid potassium doped to 500-ppm-by-weight oxygen level at 920 K is about 120 ppm by weight. The extrapolation of the solubility data reported in this study (fig. 3(a)) shows that the solubility value for molybdenum in liquid

potassium (containing less than 15 ppm by weight of oxygen) at about 920 K is about 4 ppm by weight. This low value at 920 K and the relatively small change in the solubility values of the molybdenum in liquid potassium between 1040 and 1316 K indicate that the solubility values of molybdenum determined in this study probably have not been influenced by the extraneous effects due to oxygen. This conclusion is further supported by the data presented in tables I and III. From the data in these tables, it may be concluded that the oxygen concentration in molybdenum crucibles before and after exposure to liquid potassium was about the same. Therefore, oxygen present in molybdenum crucibles probably

TABLE III. - OXYGEN CONCENTRATIONS
IN MOLYBDENUM AND TUNGSTEN
CRUCIBLES EXPOSED TO LIQUID
POTASSIUM FOR 24 HOURS

Crucible	Exposure temperature, K	Oxygen, ppm by weight
Mo-10	1316	13
Mo-11	1316	11
W-11	1328	6
W-13	1328	6
W-9	1287	8

had little or no effect on the solubility of molybdenum in liquid potassium. The low solubility values for molybdenum, low apparent heats of solution, and the probable absence of the effect of oxygen on the solubility of molybdenum in liquid potassium indicate that the data in table II and figure 3(a) are either the true solubility data, or relatively close to the true solubility data, of molybdenum in liquid potassium between 1040 and 1316 K.

The concentrations of tungsten found in liquid potassium between 1055 and 1328 K are significantly higher than those of molybdenum (figs. 3(a) and (b) and table II). Lower tungsten solubility values have been reported by Eichelberger and others (ref. 6). For example, the solubility of tungsten in liquid potassium at 1476, 1664, and 1856 K is reported to be 76, 40, and 61 ppm by weight, respectively. Thus these data suggest either that the solubility of tungsten in liquid potassium decreases with temperatures increase or that the solubility of tungsten in liquid potassium does not change with temperature increase. The solubility decrease with temperature increase or the lack of change in the solubility with temperature increase is very unlikely to occur and, therefore, the data in reference 6 are believed to be characteristic only of the experimental system used.

Using data obtained in this study, it was found that the calculated apparent heat of solution for tungsten in liquid potassium is more than twice as high as the calculated heat of solution for molybdenum in liquid potassium. The higher apparent heat of solution for tungsten in liquid potassium (17.6 kcal/mole or 73.7 kJ/mole) could mean that a chemical reaction occurred and a ternary oxide was formed from tungsten, oxygen, and potassium. This high apparent heat of solution, compared to that of molybdenum, suggests caution in accepting the solubility data presented in figure 3(b) and table II as the true solubility data for tungsten in liquid potassium between 1055 and 1328 K. It is possible that the solubility values in figure 3(b) were affected by the interstitial impurities and particularly by the oxygen. However, no studies have been made on the effects of interstitial impurities and particularly the effect of oxygen on the solubility of tungsten in liquid potassium. But the author believes that the effect of oxygen present in the tungsten crucibles probably had only a small influence on the solubility data of tungsten in liquid potassium. This can be seen from tables I and III. From the data in these tables, it probably is safe to conclude that the oxygen concentration in tungsten crucibles before and after exposure to liquid potassium was about the same. Therefore, the effect of oxygen present in tungsten crucibles on the solubility data of tungsten in liquid potassium (fig. 3(b) and table II) was probably very small. To resolve this uncertainty about the solubility data presented in this report (fig. 3(b)), studies on the possible effects of interstitials and particularly the effect of oxygen on the solubility of tungsten in liquid potassium are needed.

CONCLUSIONS

The solubilities of molybdenum and tungsten were studied in liquid potassium containing less than 15 ppm by weight of oxygen. Mathematical relations for the molybdenum and tungsten solubilities in liquid potassium were derived by the least-squares method. Conclusions that may be drawn from this study are

1. The solubility of molybdenum in high-purity liquid potassium between 1040 and 1316 K is described by the relation: $\log \text{wt ppm Mo} = (2.21 \pm 0.13) - (1472 \pm 156)/T$. At 1040 and 1316 K the solubility of molybdenum is about 6 and 13 ppm by weight, respectively; heat of solution is 6.7 ± 0.8 kilocalories per mole (28.0 kJ/mole). Chemical reactions were probably not involved in the solution process of molybdenum in liquid potassium because of the low solubility values, low heat of solution, and the probable absence of an effect of oxygen on the solubility of molybdenum in liquid potassium.
2. The solubility of tungsten in high-purity liquid potassium between 1055 and 1328 K is described by the relation: $\log \text{wt ppm W} = (5.02 \pm 0.21) - (3851 \pm 243)/T$. At 1055 and 1328 K the solubility of tungsten is about 20 and 130 ppm by weight, respectively; the ap-

parent heat of solution is 17.6 ± 1.2 kilocalories per mole (73.7 kJ/mole). The high apparent heat of solution, compared to that of molybdenum, suggests that the solubility data presented in this report might not be the true solubility of tungsten in liquid potassium. Interstitial impurities could have had some effect on the solubility of tungsten. Studies of the possible effects of interstitial impurities and particularly the effect of oxygen on the solubility of tungsten are needed to resolve this uncertainty.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 18, 1969,
129-03.

APPENDIX - PREPARATION OF CRUCIBLES AND CAPSULES

The molybdenum and tungsten crucibles, as well as the nickel capsules, were first ultrasonically cleaned in a fluorinated hydrocarbon and then ultrasonically cleaned in a detergent. After this, each metal was cleaned differently; these procedures are described below.

Molybdenum

Molybdenum crucibles, after being cleaned in a detergent at room temperature, were placed for 2 to 5 minutes in a solution of 77 parts of 70-percent-by-weight nitric acid and 23 parts of distilled water. Afterward, the crucibles were cleaned for 2 minutes in a solution made of 27 parts of 37-percent-by-weight hydrochloric acid and 73 parts of distilled water. Then the crucibles were rinsed several times with distilled water and dried in a desiccator under vacuum.

Tungsten

Tungsten crucibles, after being cleaned in a detergent at room temperature, were immersed for 5 minutes in a boiling potassium hydroxide solution (20 percent by weight). The crucibles were removed, rinsed with distilled water and etched at 333 K for 15 minutes in a solution of 21 parts of 70-percent-by-weight nitric acid, 4 parts of 49-percent-by-weight hydrofluoric acid, and 75 parts of distilled water. After this, the crucibles were rinsed several times with distilled water and dried in a desiccator under vacuum.

Nickel

Nickel capsules, after being cleaned in a detergent at room temperature, were etched at 363 K for 1/2 to 1 minute in a solution of 30 parts of 70-percent-by-weight nitric acid, 10 parts of concentrated orthophosphoric acid, 10 parts of concentrated sulfuric acid, and 50 parts of glacial acetic acid. After this, the capsules were rinsed several times with distilled water and dried in a desiccator under vacuum.

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